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**COMPARISON OF THE TRIBOLOGICAL PROPERTIES AT 25° C
OF SEVEN DIFFERENT POLYIMIDE FILMS BONDED TO
301 STAINLESS STEEL**

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COMPARISON OF THE TRIBOLOGICAL PROPERTIES
AT 25°C OF SEVEN DIFFERENT POLYIMIDE FILMS
BONDED TO 301 STAINLESS STEEL

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SUMMARY

The tribological properties of seven different types of polyimide films bonded to 301 stainless steel substrates were studied at 25°C using a pin-on-disk friction and wear apparatus. In order to reduce the stress on the film, a hemispherically-tipped pin with a 1.20 mm diameter flat was slid against the films. Under a 1-Kg load the projected stress on the films was 8.6×10^3 kPa (1250 psi). Even with this low stress (as compared to a hemisphere sliding on the film) all the films brittlely fractured and spalled from the wear track in less than 1000 cycles of sliding.

The spalling was believed to be caused by the film's inability to withstand the tensile stresses imparted by localized deformations (misalignment, edge effects and asperity interactions) of the 301 substrate. This implies a hard substrate is a necessary prerequisite for good lubrication with polyimide films. A second possibility for the spalling might be that the films did not have good adherence to the 301 stainless steel substrate.

Since the substrate properties dominated the results, this study did not give a true disclosure as to which (if any) polyimide film would provide the best tribological results. Three polyimides (PIC-1, PIC-2 and PIC-6) did not spall as badly as the others. Thus, they may have slightly better tensile properties than the others and may provide better results when applied to harder substrates. The friction coefficient results for six of the polyimides varied between 0.21 to 0.32, while one polyimide (PIC-6) varied from 0.32 to 0.39.

INTRODUCTION

It has been shown in previous investigations (refs. 1 and 2) that polyimide and polyimide-bonded graphite fluoride films have potential for solid

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lubricant applications where long thermal soaks are encountered. Low weight loss rates, good adhesion and good friction and wear properties were obtained for films thermally exposed at temperatures up to 315°C.

The word polyimide is a generic designation and refers to a class of long-chain polymers which have recurring imide groups as an integral part of the main chain. By varying the monomeric starting materials, polyimides of different chemical composition and structure can be obtained.

In previous tribological studies (refs. 1 to 9), a commercially available polyimide designated PI-4701 was used. There are now commercially available many other polyimides. The object of this investigation was to determine which of these polyimides had the best friction and wear properties at 25°C.

Seven different commercially available polyimides were evaluated. They were designated PIC-1 through PIC-7. PIC-1 was the polyimide used in previous investigations (refs. 1 to 9). The films were applied to 301 stainless steel disks and evaluated on a pin-on-disk type of friction and wear apparatus. The pin material was 440C HT stainless steel and was slid against the film under a 1-Kg load at a speed of 1000 rpm (2.6 meters per second). Instead of sliding a hemispherically-tipped rider (as is normally done), a rider with a 1.20 mm diameter flat on it was slid against the film. This was done so that a controlled, lower, constant contact stress could be achieved.

MATERIALS

The polyimides used were obtained as precursor solutions. In most instances a thinner consistency of N-methyl-pyrrolidone and xylene was added to the polyimides to make them sprayable. Seven different types of condensation polyimides were evaluated. The chemical composition and structure of five of the condensation polyimides were proprietary. They are designated in Table I as polyimide types PIC-1 to PIC-5. The structures of the other two polyimides are known and are shown in Figures 1 and 2. They are designated as polyimide types PIC-6 and PIC-7 in this investigation. The polyimide used in previous work by this investigator (refs. 1 to 10) is designated as PIC-1. The films were applied to AISI 301 stainless steel disks (1.2 cm thick by 6.3 cm diameter) that had a Rockwell hardness of B-87. The riders used were made of AISI 440C HT stainless steel of Rockwell hardness C-58.

FRICTION APPARATUS

A conventional type of pin-on-disk friction and wear apparatus was used in this study (fig. 3); except that prior to conducting the experiments, a 1.20 millimeter flat was worn onto the 0.476 cm-radius hemispherically-tipped rider (pin). A 1-kilogram load was applied to the flat on the rider which gave a projected contact stress of 8.6×10^3 kPa (1250 psi) against the film. The disk was 6.3 centimeter in diameter and the rider slid on the disk at a radius of 2.5 centimeters. The disk was rotated at 1000 rpm which gave it a linear sliding speed of 2.6 meters per second. The friction specimens were enclosed in a chamber in order that the atmosphere could be controlled. To obtain the controlled air atmosphere of 10 000 ppm, H_2O (approximately 50 percent R.H. at $25^\circ C$), dry air and dry air bubbled through water were mixed.

PROCEDURE

Generation of Rider Flat

The flat on the 0.476 cm-radius hemispherically-tipped rider (pin) was generated prior to conducting the friction and wear tests by sliding it against a rubbed graphite fluoride film which was applied to a sand-blasted AISI 440C HT stainless steel disk. The rider was not removed from the holder after the flat was generated or while it was cleaned. The rider and the disk (with applied polyimide film) were positioned (and indexed) in the apparatus by using a linear variable differential transformer (LVDT) so that a flat-on-flat configuration was produced with minimal misalignment (fig. 3).

Surface Preparation and Cleaning

The disk surfaces were roughened by sanding with number 150 wet sandpaper to a cla roughness of 0.25 to 0.36 micrometers. After surface roughening, the disks were scrubbed with a brush under running tap water to remove the abrasive particles. A water paste of levigated alumina was next rubbed over the surface with a polishing cloth. This was followed by a second scrubbing under running tap water. The disks were rinsed in distilled water and then clean, dry compressed air was used to quickly dry the surfaces. The disks were stored in a desiccator until they were ready for coating with the solid lubricant.

Film Application

An artist's airbrush was used to apply the polyimide films to the disks. The films did not dry rapidly; thus, only a thin layer was

applied at one time in order to prevent "running." Each layer was completely cured before the next layer was applied. The cure was to raise the oven temperature from 25°C to 75°C and hold one-half hour, from 75°C to 125°C and hold one-half hour, from 125°C to 175°C and hold one-half hour, from 175°C to 225°C and hold one-half hour, and from 225°C to 300°C and hold three-quarters of an hour. The temperature was then held at 300°C for 1 hour. The film thickness was measured after each cure to determine if another layer was necessary to achieve the desired thickness.

The polyimides used in this study are not normally applied as thin films; thus, the curing procedure (as given by the manufacturer) was not fully specified. The basic curing theory is to drive the H₂O and solvent out of the precursor solution at a low temperature before polymerization takes place at a higher temperature. Polymerization is time and temperature dependent. The higher the temperature, the shorter the time needed to achieve the same degree of polymerization. In previous studies on PIC-1 polyimides (refs. 1 to 9), the cure procedure was to bake at 100°C for 1 hour and at 300°C for 2 hours. This cure did not work on PIC-7 polyimide; the films were found to be cracked and blistered. To obtain a good film with PIC-7 polyimide, it was necessary to raise the temperature more slowly from room temperature. Thus, the previously mentioned cure schedule was devised to obtain good PIC-7 films. The same cure was tried on the six other condensation polyimides and good films were also obtained. Therefore, this cure schedule was used as the standard in this study.

Friction and Wear Tests

The procedure for conducting the friction and wear tests was as follows: A rider and disk (with applied solid lubricant film) were inserted into the friction apparatus and the test chamber was sealed. Moist air (10 000 ppm H₂O) was purged through the chamber for 15 minutes. Moist air was used as a controlled atmosphere. The flow rate was 1500 cubic centimeters per minute, and the volume of the chamber was 2000 cubic centimeters. After purging for 15 minutes, the disk was set into rotation at 1000 rpm and a 1-kilogram load was gradually applied. The test temperature was 25°C.

Each test was stopped after 1000 cycles of sliding. After the rider and disk were removed from the friction apparatus, the contact areas were examined by optical microscopy and photographed. Surface profiles of the disk wear tracks were also taken. For some tests the rider and disk were placed back into the apparatus and the previous test procedure was repeated. The rider was not removed from the holder, and locating pins in the apparatus insured that it was returned to its original position. The same was true for the disk.

RESULTS AND DISCUSSION

Friction Coefficient

Representative friction traces for the 1.20-millimeter diameter flat sliding against films of the seven different polyimides are shown in Figures 4 to 10. The load was applied gradually to the rider and the rotating disk; thus, the friction trace started out at zero and increased with the number of sliding revolutions until the full load was applied. It took 50 to 75 cycles of sliding before the full load was applied.

The friction traces are different for each polyimide, but in general tend to level off after the full load was applied at a value of between 0.24 to 0.32. The one exception was PIC-6 which leveled off at a value of between 0.34 to 0.39 (fig. 9). The friction coefficient of two polyimides, PIC-3 and PIC-4, increased markedly after 750 cycles of sliding to a value greater than 0.50 (figs. 6 and 7).

Rider and Film Wear

Film wear was studied by taking surface profiles and by observing the film wear track area with an optical microscope. Figure 11 gives representative surface profiles of the wear tracks on the seven different types of polyimide films after 1000 cycles of sliding. Figures 12 to 18 give photomicrographs of the rider contact areas and the film wear track areas for the same sliding interval.

The surface profiles and photomicrographs indicate that after 1000 cycles of sliding the films had been worn through to the metallic substrate. The films did not all wear in the same manner, however. PIC-1 and PIC-6 tended to wear in a series of spalls or pits which extended around the circumference of the track. In between the spalls, the film was still intact and only slight wear occurred (figs. 12(b) and 17(b)).

Polyimide films PIC-3, PIC-4, PIC-5 and PIC-7 were found to be completely spalled from the wear track area (figs. 14(b), 15(b), 16(b) and 18(b)). Galling wear and transfer were found to occur on the riders which slid on polyimide films PIC-3, PIC-4 and PIC-5 (figs. 14(a), 15(a) and 16(a)). No galling wear was found with the PIC-7 film (after 1000 cycles of sliding) and a thin layer of the polyimide was still present on the rider contact area and on the wear track (fig. 18).

The PIC-2 film also spalled (fig. 13(b)), but the spalls were not as predominant as with the other polyimides and the film seemed to possess more plasticity than the others. The surface profiles of the wear tracks shown in Figure 11 indicate that the PIC-2 film plastically flowed at the slides of the wear track to a greater extent than did the

other seven polyimide films. Also, no galling wear or transfer were observed on the rider which slid on the PIC-2 film.

Polyimide films PIC-1, PIC-2, PIC-6 and PIC-7 prevented galling wear to the surfaces for 1000 cycles of sliding. However, on continued sliding, failure was eventually marked by galling wear. For example, Figure 19 shows the rider contact area and film wear track area for the rider sliding on the PIC-6 film after 15 000 cycles. Both surfaces are characterized by a galling type wear. No wear of the 440C HT stainless steel riders was observed, but the 301 stainless steel substrate transferred to the rider in what appears to be an adhesive type of wear mechanism.

Concluding Remarks

In previous studies (refs. 8 and 9) on PIC-1 polyimide films applied to AISI 440C HT stainless steel disk substrates of Rockwell hardness C-58, it was found that the film wear process (for a 0.476 cm-radius hemispherically-tipped pin sliding on the film) was one of gradual wear through the film (fig. 20). It took approximately 60 000 cycles of sliding (under identical experimental conditions to those used in this investigation) to wear through a 0.0025 cm thick film.

When a hemisphere was slid against the PIC-1 films applied to AISI-301 stainless steel substrates, the films brittlely fractured and failed immediately. In reference 10, it was shown that the film wear rate of polyimide (PIC-1) bonded graphite fluoride films was reduced about 700 times when a 0.95 mm diameter flat area was slid against the films rather than a hemisphere. This reduction in wear rate appeared to be due to a reduction of contact stress. High stresses initiated crack propagation within the film and caused spalls to occur.

For the above reason, a flat was slid against the various polyimide films in this study rather than a hemisphere. Nevertheless, the films still brittlely fractured and spalled. This could have been caused by poor adherence between the polyimide and the 301 stainless steel substrate. However, bending tests on PIC-1 films bonded to 304 stainless steel foils in references 1 and 2 indicated that the films had good adherence.

A more likely reason for the cracking could have been due to the fact that 301 stainless steel is much softer than 440C HT stainless steel (Rockwell B-87 compared to C-58). The yield strength of 301 is 2.7×10^5 kPa (40 000 psi) compared to 1.9×10^6 kPa (275 000 psi) for 440C (ref. 11). Even though the average projected contact stress for the rider sliding on the film was 8.6×10^3 kPa (1250 psi), slight misalignment, edge effects or asperity interactions could have led to much

higher stresses under sliding conditions. Polyimide is a rather brittle polymer and does not have good tensile properties. Apparently, the soft 301 stainless steel sufficiently deformed locally, under the conditions of these experiments, to cause a tensile fracture of the films under sliding conditions.

The results indicate that the substrate type can play a major role in the tribological performance of polyimide films.

SUMMARY OF RESULTS

Friction and wear studies on seven different types of polyimide films applied to 301 stainless steel substrates indicate that:

1. The substrate material (to which the polyimide films were applied) dominated the results and a true disclosure of which polyimide was best could not be determined.
2. All seven polyimide films spalled in less than 1000 cycles of sliding. Either the films were not able to withstand the tensile stresses (under sliding conditions) imparted by the deformation of the soft substrate or the films did not have good adherence to the 301 stainless steel substrate.
3. For the first 1000 cycles of sliding, the friction coefficient for six of the films varied between 0.21 to 0.32. One (PIC-6) varied from 0.32 to 0.39.
4. Three polyimides (PIC-1, PIC-2 and PIC-6) did not spall as badly as the others. Thus, they may have slightly better tensile properties than the others.

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TABLE 1. - DESIGNATION OF TYPES OF POLYIMIDES
USED IN THIS INVESTIGATION

<u>Designation</u>	<u>Type</u>	<u>Structure</u>
PIC-1	Condensation	Proprietary
PIC-2	Condensation	Proprietary
PIC-3	Condensation	Proprietary
PIC-4	Condensation	Proprietary
PIC-5	Condensation	Proprietary
PIC-6	Condensation	Figure 1
PIC-7	Condensation	Figure 2

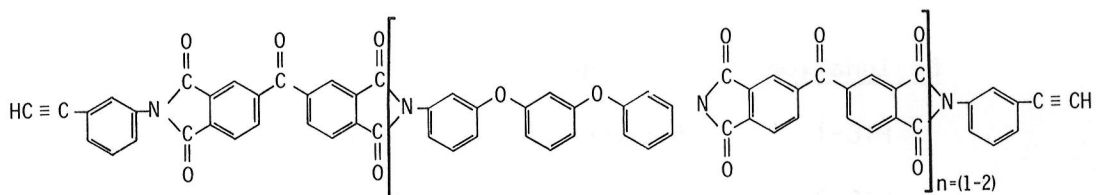


Figure 1. - Structure of polyimide PIC-6.

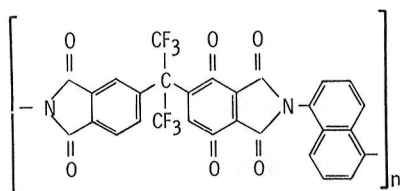


Figure 2. - Structure of polyimide PIC-7.

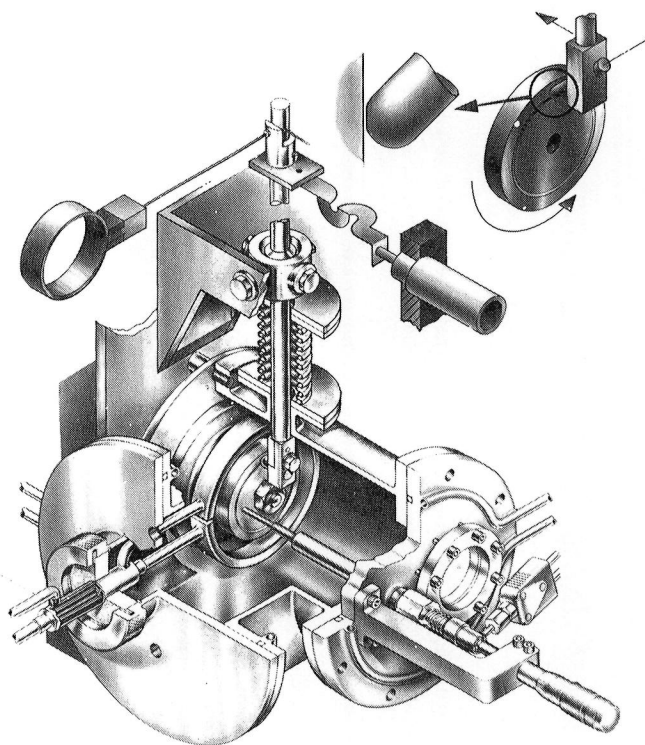


Figure 3. - Pin-on-disk friction and wear apparatus.

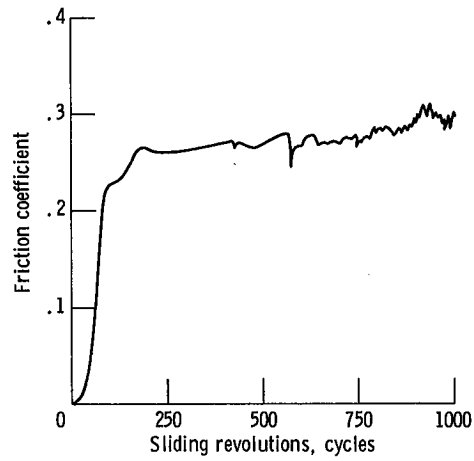


Figure 4. - Friction trace for a 440C HT stainless steel rider (with 1.20-mm diameter flat) sliding on a PIC-1 polyimide film bonded to a 301 stainless steel disk.

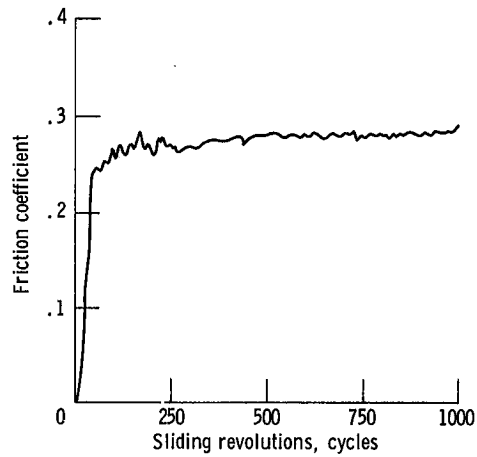


Figure 5. - Friction trace for a 440C HT stainless steel rider (with 1.20-mm diameter flat) sliding on a PIC-2 polyimide film bonded to a 301 stainless steel substrate.

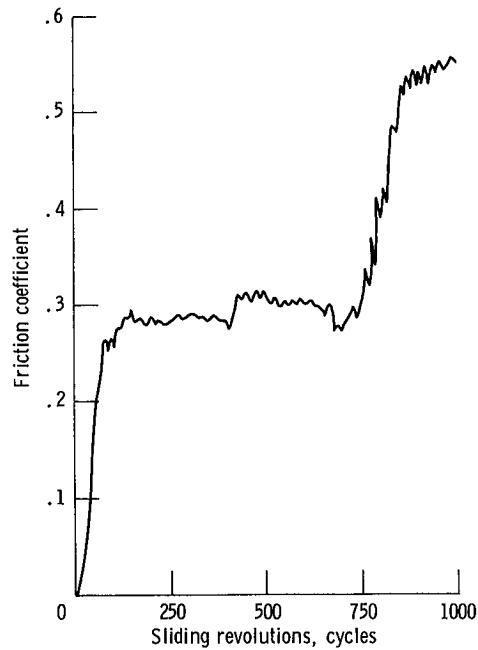


Figure 6. - Friction trace for a 440C HT stainless steel rider (with 1.20-mm diameter flat) sliding on a PIC-3 polyimide film bonded to a 301 stainless steel disk.

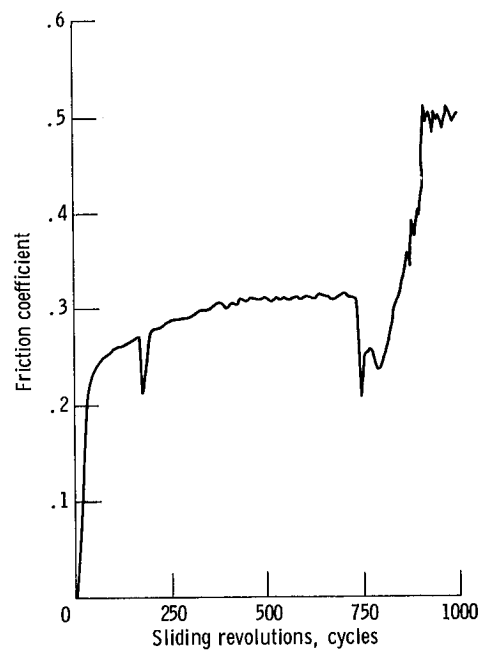


Figure 7. - Friction trace for a 440C HT stainless steel rider (with 1.20-mm diameter flat) sliding on a PIC-4 polyimide film bonded to a 301 stainless steel disk.

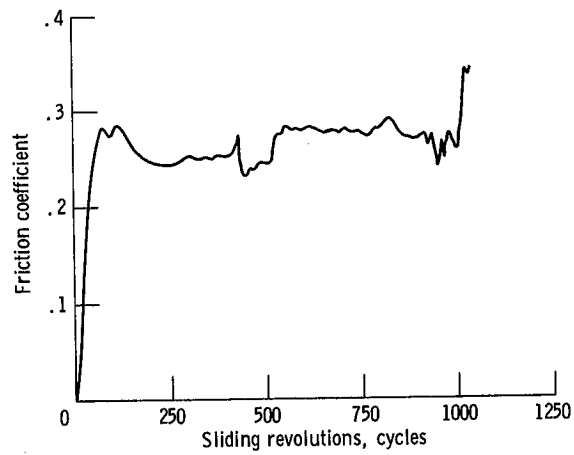


Figure 8. - Friction trace for a 440C HT stainless steel rider (with 1.20-mm diameter flat) sliding on a PIC-5 polyimide film bonded to a 301 stainless steel disk.

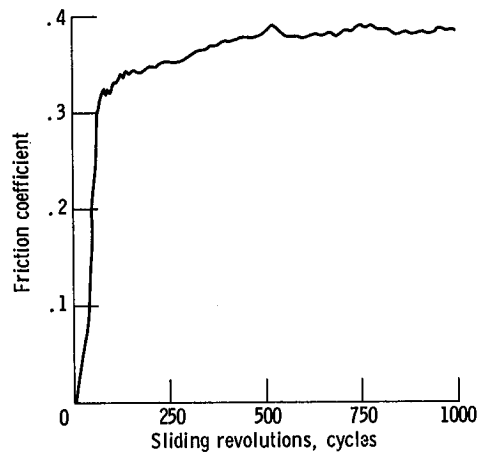


Figure 9. - Friction trace for a 440C HT stainless steel rider (with 1.20-mm diameter flat) sliding on a PIC-6 polyimide film bonded to a 301 stainless steel disk.

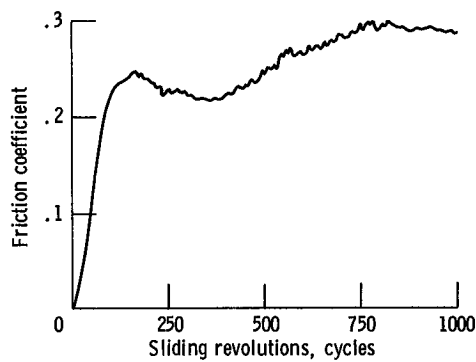


Figure 10. - Friction trace for a 440C HT stainless steel rider (with 1.20-mm diameter flat) sliding on a PIC-7 polyimide film bonded to a 301 stainless steel disk.

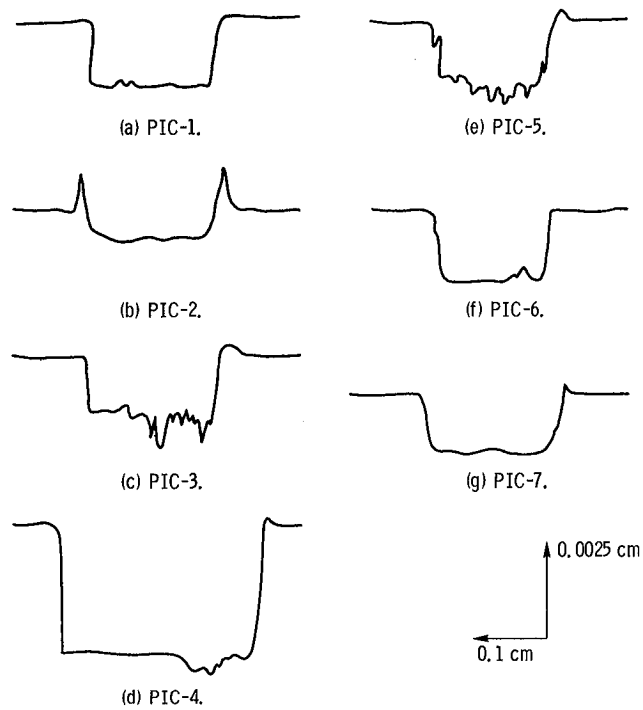
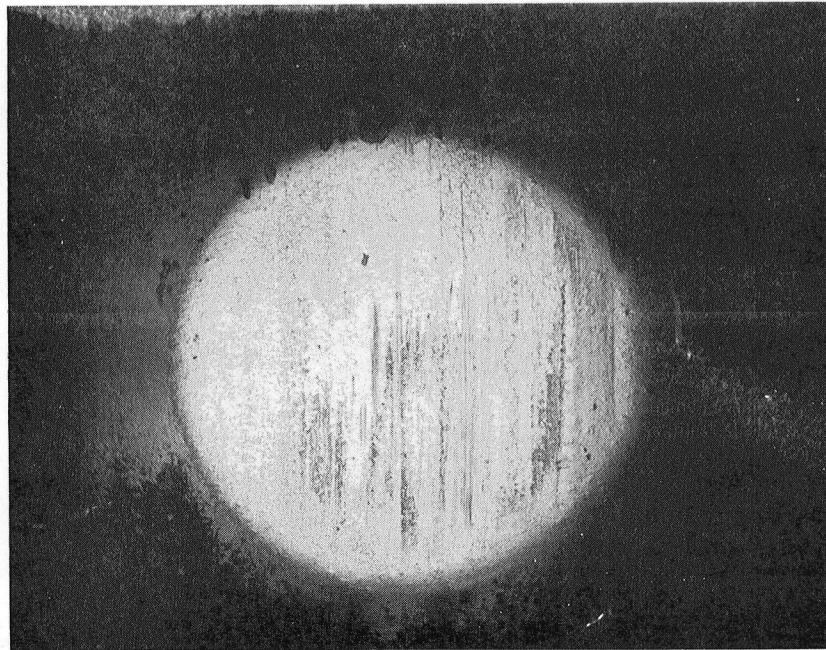


Figure 11. - Surface profiles of the wear tracks on seven different types of polyimide films after 1000 cycles of sliding.

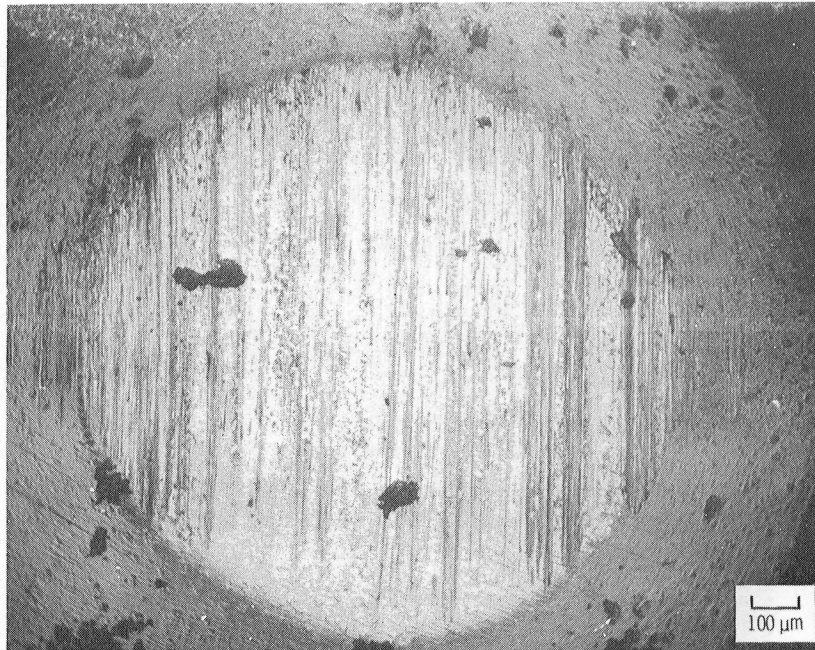


(a) Rider contact area.

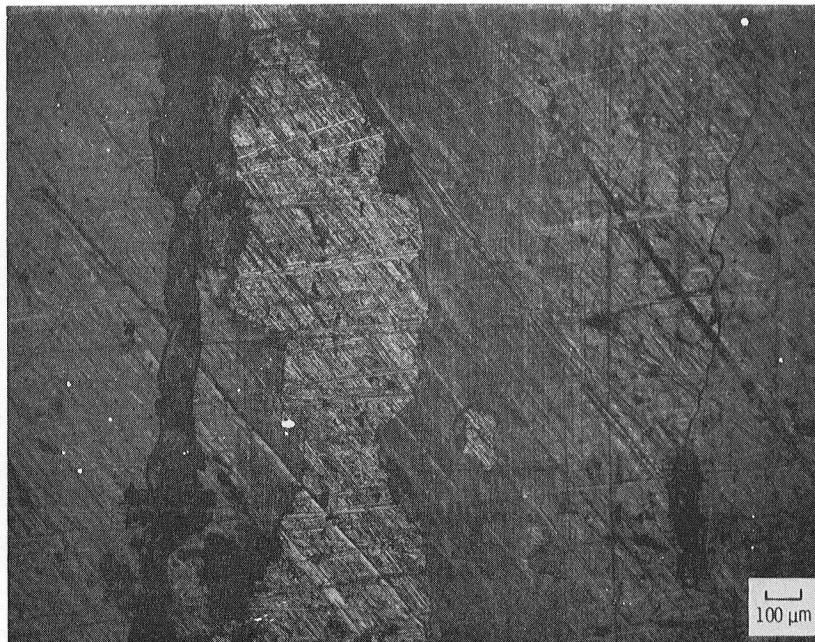


(b) Film wear track area.

Figure 12. - Photomicrographs of the contact areas after 1000 cycles of sliding for a 440C HT stainless steel rider sliding on a PIC-1 polyimide film bonded to a 301 stainless steel disk.



(a) Rider contact area.

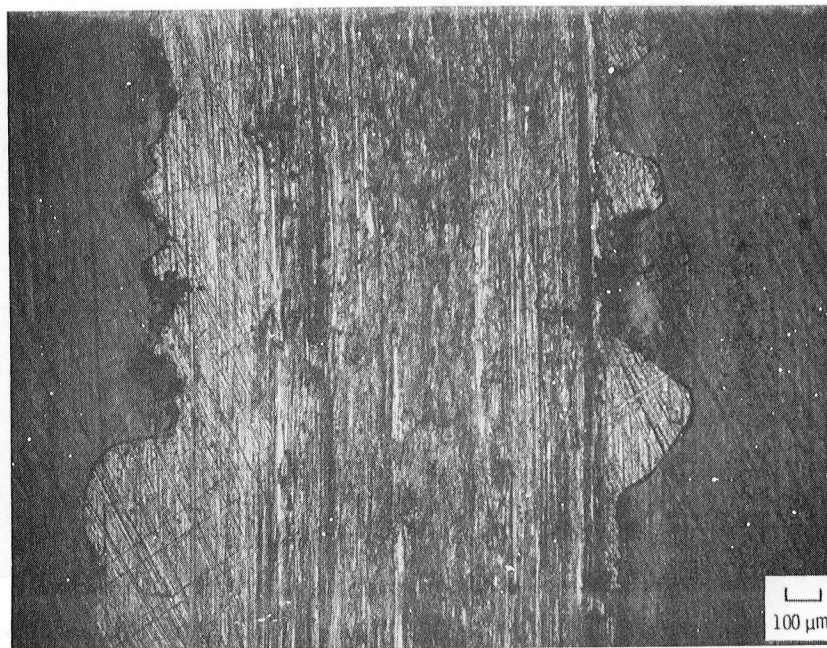


(b) Film wear track area.

Figure 13. - Photomicrographs of the contact areas after 1000 cycles of sliding for a 440C HT rider sliding on a PIC-2 polyimide film bonded to a 301 stainless steel disk.

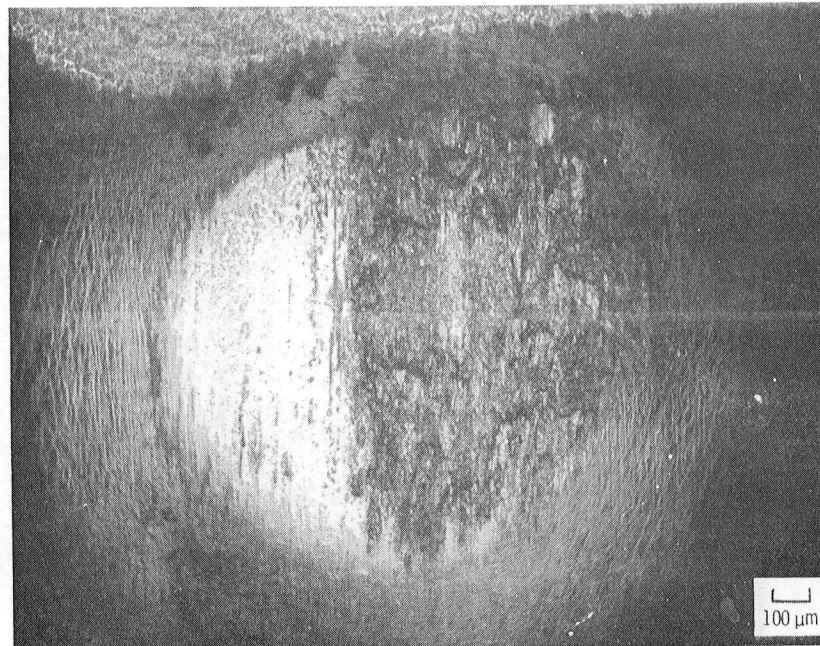


(a) Rider contact area.

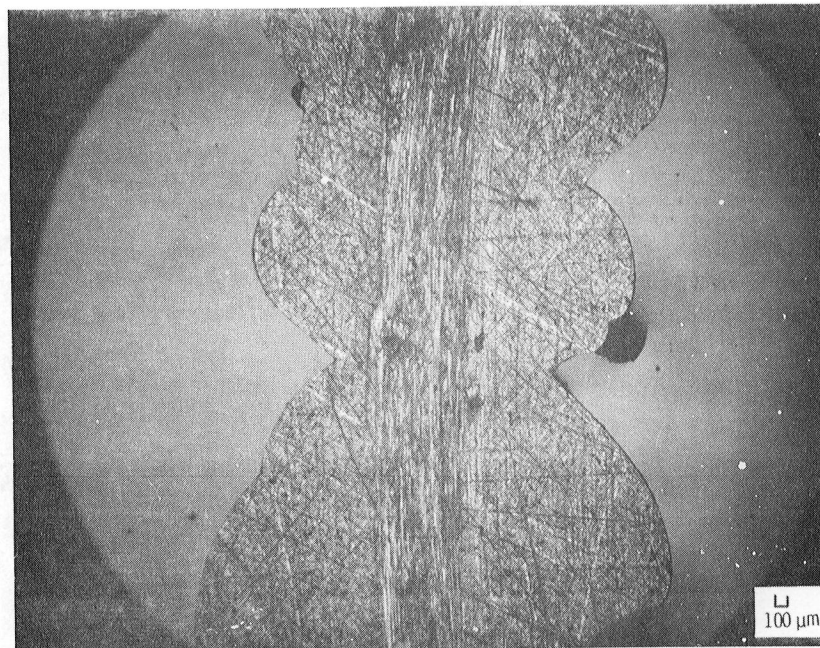


(b) Film wear track area.

Figure 14. - Photomicrographs of the contact areas after 1000 cycles of sliding for a 440C HT rider sliding on a PIC-3 polyimide film bonded to a 301 stainless steel disk.

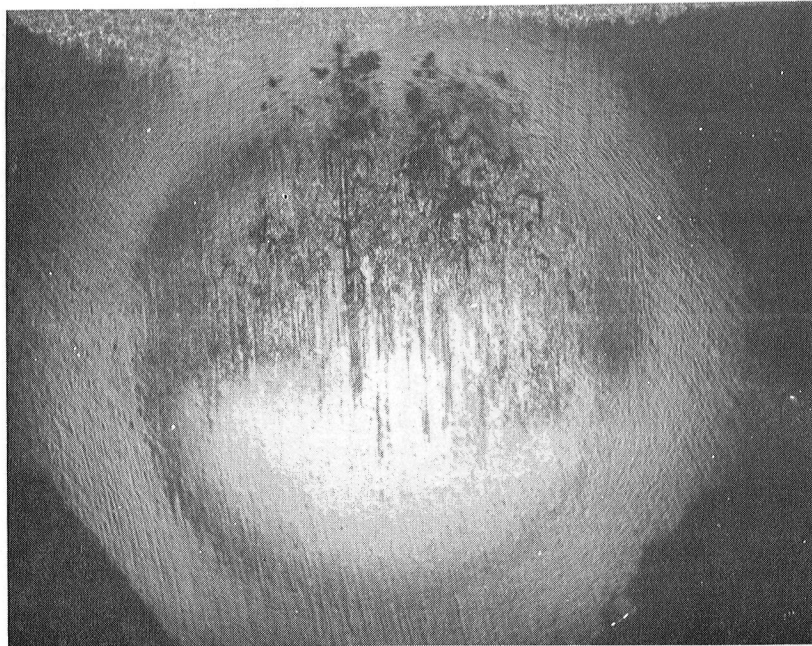


(a) Rider contact area.



(b) Film wear track area.

Figure 15. - Photomicrographs of the contact areas after 1000 cycles of sliding for a 440C HT rider sliding on a PIC-4 polyimide film bonded to a 301 stainless steel disk.



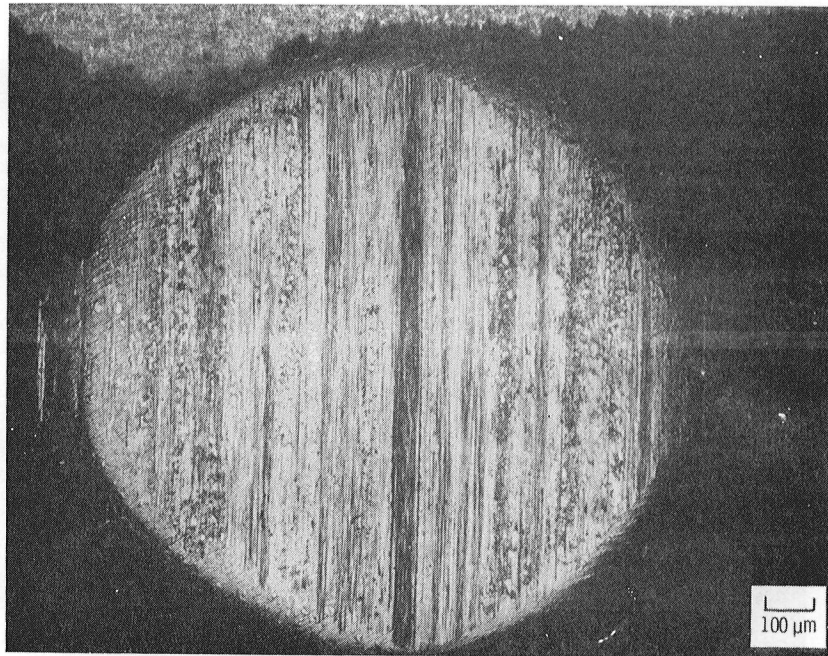
(a) Rider contact area.



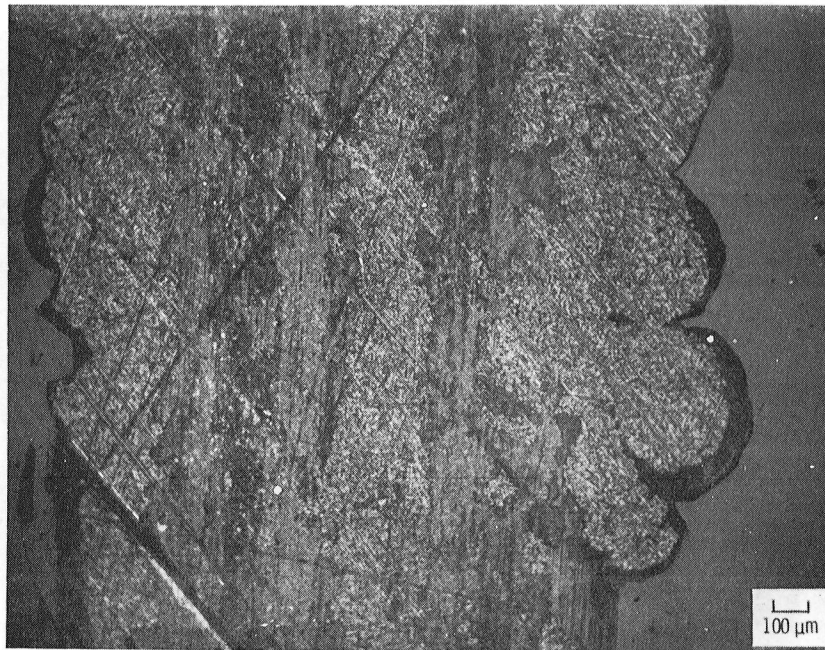
(b) Film wear track area.

Figure 16. - Photomicrographs of the contact areas after 1000 cycles of sliding for a 440C HT rider sliding on a PIC-5 polyimide film bonded to a 301 stainless steel disk.

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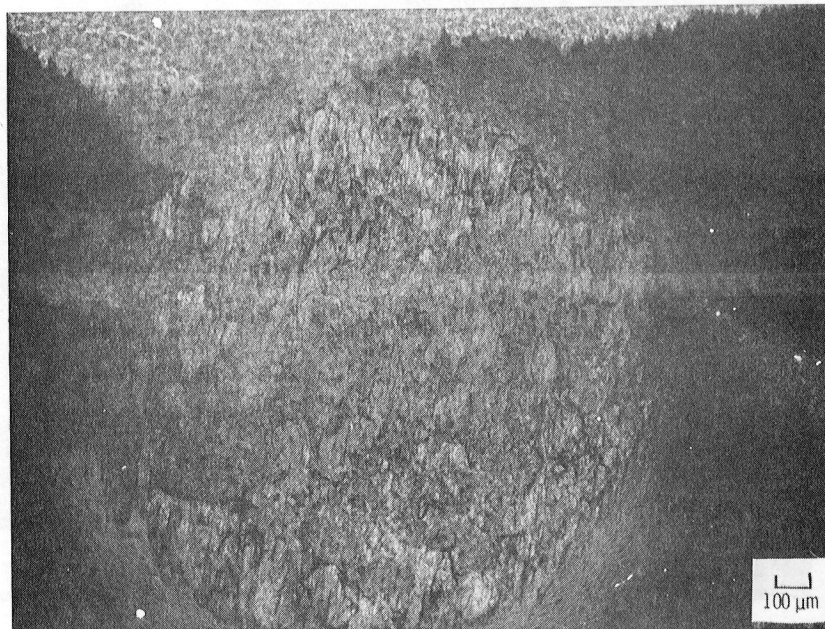


(a) Rider contact area.

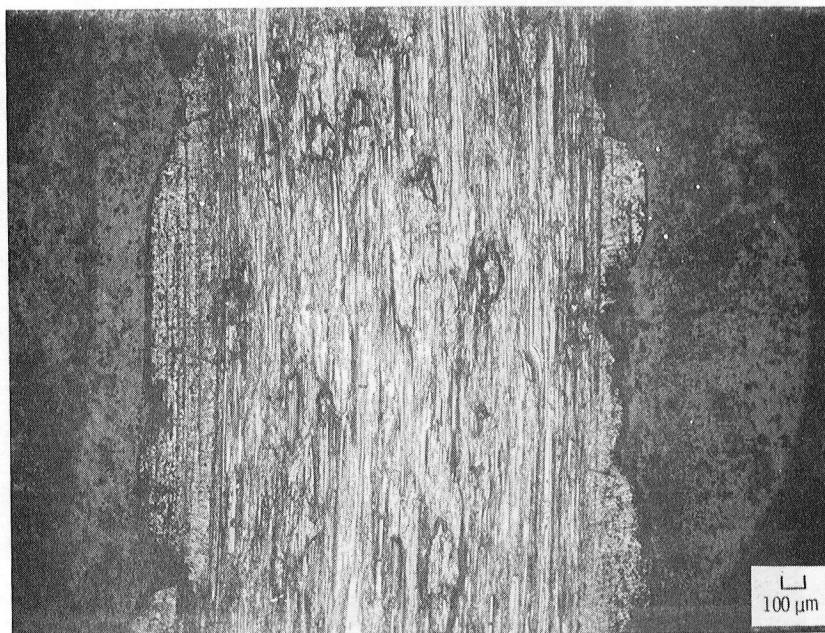


(b) Film wear track area.

Figure 18. - Photomicrographs of the contact areas after 1000 cycles of sliding for a 440C HT stainless steel rider sliding on a PIC-7 polyimide film bonded to a 301 stainless steel disk.



(a) Rider contact area.



(b) Film wear track area.

Figure 19. - Photomicrographs of the contact areas after 15 000 cycles of sliding for a 440C HT stainless steel rider sliding on a PIC-6 polyimide film bonded to a 301 stainless steel disk.

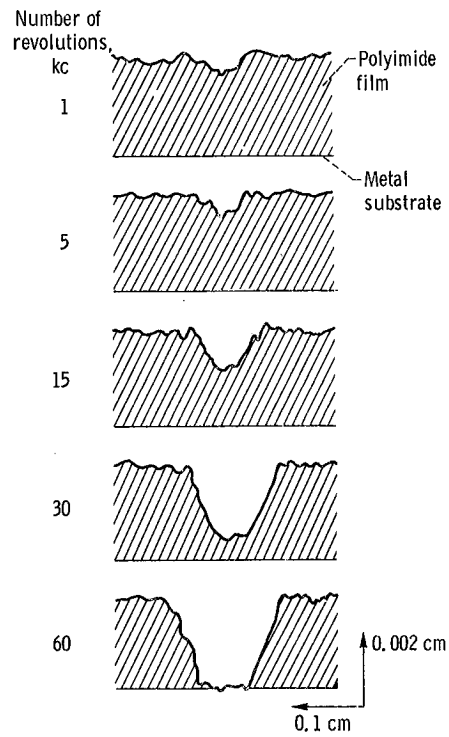


Figure 20. - Surface profiles of the wear track on a polyimide film (PIC-1) bonded to a 440C HT stainless steel disk after various sliding intervals. Sliding speed, 2.6 meters per second; load, 1 kilogram; temperature, 25° C; 440C HT stainless steel rider. (Data from ref. 8.)

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